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POLYPHENYLQUINOXALINES FROM HIGHLY FUSED TETRAAMINES

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TECHNICAL REPORT AFML-TR-72-285

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FOREWORD

This report was prepared by the Polymer Branch, Nonmetallic Materials Division. The work was initiated under Project No. 7340, "Nonmetallic and Composite Materials". It was administered under the direction of the Air Force Materials Laboratory, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio, with Dr. F. E. Arnold as Project Scientist.

This report covers work conducted from June 1971 to September 1972. The manuscript was released by the authors in September 1972 for publication as a technical report.

This technical report has been reviewed and is approved.



R. L. VAN DEUSEN
Chief, Polymer Branch
Nonmetallic Materials Division
Air Force Materials Laboratory

ABSTRACT

A series of polyphenylquinoxalines has been synthesized by the reaction of p,p'-oxydibenzil with aromatic tetraamines containing varying numbers of fused rings. In addition, it was found that the glass transition temperatures of the resultant polymers increased as the number of adjacent fused rings in the polymers was increased. This provided the basis for achieving glass transition temperatures for the polymers in excess of 400°C, substantially higher than the state-of-the-art polyphenylquinoxalines. All of the polymers displayed both the good thermal stability and the solubility in m-cresol characteristic of the polyphenylquinoxaline family of polymers.

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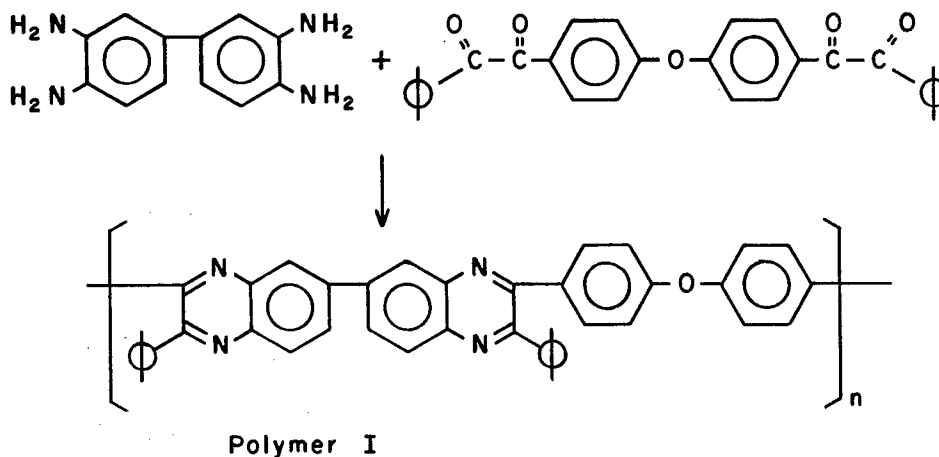
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SECTION I

INTRODUCTION

Materials requirements for light-weight, high-temperature composites and adhesives for Air Force structural applications have led to the exploration of a number of polymeric systems which might be utilized in such materials. One of the promising families of candidate polymers has been the aromatic heterocyclic polyphenylquinoxalines (PPQ's) (References 1, 2, 3, 4, and 5). These polymers are generally prepared by the solution polycondensation of an aromatic bis(o-diamine) such as 3,3'-diaminobenzidine with a bis(phenyl- α -dicarbonyl) compound such as p,p'-oxydibenzil.

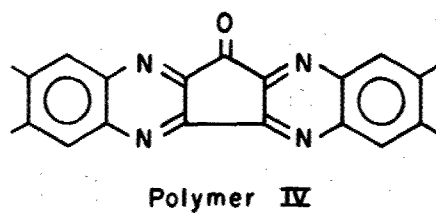
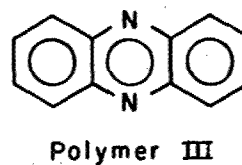
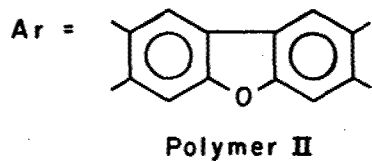
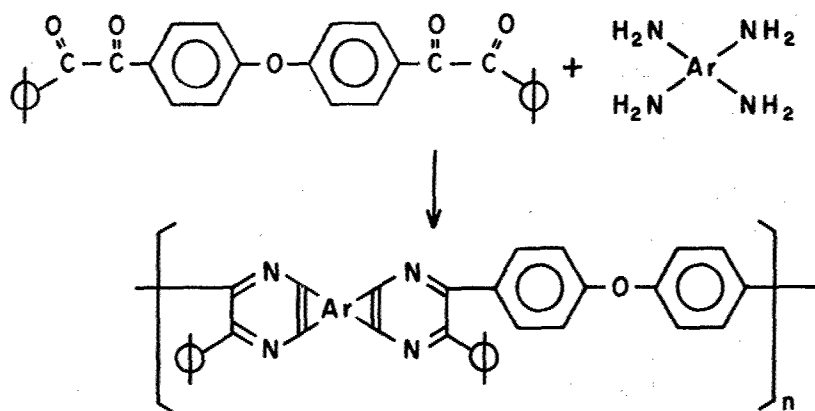


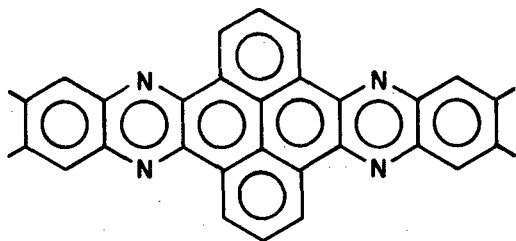
The PPQ polymers exhibit good thermal stability, with a decomposition temperature for some samples of greater than 500°C, as determined by TGA. A desirable feature of the PPQ polymers is their solubility in aprotic solvents, thus making them amenable to processing. However, a limiting factor in the use of the PPQ's has been the relatively low glass transition temperatures (Tg's) found for these polymers. For example, a Tg of 284° was reported from DSC examination of polymer I (Reference 5). With a goal of increasing the Tg while retaining the solubility in aprotic solvents, we have explored the effect on PPQ's of increasing the number of adjacent fused rings in the tetraamine monomer used. This report contains the results of this exploratory study.

SECTION II

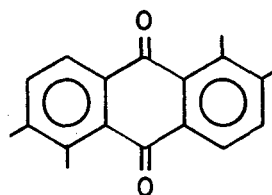
RESULTS AND DISCUSSION

Five different aromatic heterocyclic fused tetraamines were condensed with p,p'-oxydibenzil to give polymers II, III, IV, V, and VI.





Polymer V



Polymer VI

The polymerizations were carried out in m-cresol from room temperature to 180°C. The polymers were isolated by precipitation into anhydrous methanol, and purified by analogous reprecipitation techniques.

All the polymers displayed the desired solubility in m-cresol. A tabulation of viscosity and Tg data is given in Table I for polymers II-VI, with the literature data for polymer I included for comparison. With the exception of VI, the polymers are arranged in order of increasing number of adjacent fused rings. It can be seen from Table I that the desired increase in Tg was obtained in going from polymer I to the more highly fused polymer II. The onset of decomposition for polymers III-VI in nitrogen prevented any further correlation of Tg with increasing number of fused rings in these polymers.

The thermal properties of the polymers were quite good as shown by thermogravimetric analysis. Weight losses of 5 to 10% usually considered to be catastrophic, were not seen to occur for the polymers up to 600 to 650°C in nitrogen and 450 to 500°C in air. This is not surprising since the PPQ polymers have been shown to possess excellent thermal properties.

We have thus demonstrated that the Tg's of PPQ type polymers can be raised to temperatures in excess of 400°C by the incorporation of fused ring tetraamines in the backbone of the polymer. This is indicative of a substantial increase (more than 100°C) in the use temperature potential of this polymer system over state-of-the-art. Another critical aspect of these novel fused-ring PPQ's is that they retain the characteristic solubility of PPQ's in m-cresol, thus making it possible

TABLE I
INHERENT VISCOSITIES AND GLASS TRANSITION
TEMPERATURES OF HIGHLY FUSED PPQ POLYMERS

| Polymer | η_{inh} (dl/g) | T_g (°C) |
|---------|---------------------|----------------------|
| I | 1.8 ^{a,b} | 284°C ^{a,c} |
| II | 0.74 ^d | 406°C ^f |
| III | 0.32 ^d | none ^{f,g} |
| VI | 0.43 ^e | none ^{f,g} |
| IV | 0.45 ^e | none ^{f,g} |
| V | 0.43 ^d | none ^{f,g} |

a. Reference 5

b. 0.5% in H₂SO₄ at 25°C

c. Determined by dielectric loss measurements

d. 0.3% in H₂SO₄ at 30°C

e. 0.3% in m-cresol at 30°C

f. Determined by DTA at 20°/min

g. T_g not exhibited below decomposition temperature

to prepare dopes in this solvent for attempts to process these polymers in the future. The novel type of PPQ synthesized from tetraamino-dibenzofuran (polymer II) appears to have the highest potential. It was obtained in highest molecular weight and also found to have the best film-forming properties in these initial investigations.

SECTION III
EXPERIMENTAL

1. MONOMERS

p,p'-Oxydibenzil was prepared (Reference 6) under USAF Contract F33615-69-C-1351 according to the method of Hergenrother (Reference 7). The material, m.p. 108°-109°, was used as received.

2,3,7,8-Tetraaminodibenzofuran was prepared under USAF Contract F33615-69-C-1257 by a two-step nitration of dibenzofuran followed by catalytic reduction (References 8 and 9).

2,3,7,8-Tetraaminophenazine was prepared (Reference 9) under USAF Contract F33615-69-C-1257 according to the method of Nietzki and Muller (Reference 10). The monomer was analyzed and used as received.

Leuconic acid pentahydrate was prepared according to the reported procedure of Fatiadi, Isbell, and Sager (Reference 11) by stepwise oxidation of tetrahydroxybenzoquinone.

1,2-Diamino-4,5-o phenylene-bis(p-Toluenesulfonamide) benzene was prepared according to the procedure reported by Arnold (Reference 12) by the reduction of 1,2-dinitro-4,5-o-phenylene-bis(p-toluenesulfonamide)-benzene using sodium hydrosulfite in water/DMAC solvent.

Preparation of 2,3,8,9-tetra(p-toluenesulfonamido)diquinoxal(2,3-e: 2', 3'-1)cyclopentanepentone

A mixture of 6.4g (14 mmole) of 1,2-diamino;4,5-o-phenylene-bis(toluene-sulfonamide) and 1.6g (7 mmole) of leuconic acid pentahydrate (cyclopentanepentone) in 100 ml of m-cresol was heated at reflux under nitrogen for three hours. The reaction mixture was then evaporated to near dryness in vacuo, and the tarry residue dissolved in a minimum amount of acetone. The resulting solution was filtered, and the filtrate added to 200 ml of hexane, precipitating a brown solid.

The product was collected and reprecipitated from acetone by the previously described procedure. The product thus obtained was dried at 80° in vacuo, yielding 7.4g (96%) of 2,3,8,9-tetra(toluenesulfonamido)-diquinoxal-(2,3-e:2',3'-1)cyclopentanepentone as an amorphous brown powder, m.p. 245° (dec.). The infrared spectrum is shown in Figure 1.

Anal. Calcd. for $C_{45}H_{36}N_8O_9S_4$; C, 56.24; H, 3.78; N, 11.66
Found; C, 56.95 H, 3.70; N, 11.20

Preparation of 2,3,8,9-tetraminodiquinoxal(2,3-e:2',3'-1)cyclopentane-pentone

To 100 ml of de-aerated sulfuric acid was added 5.0g (5.1 mmole) of finely powdered 2,3,8,9-tetra(p-toluenesulfonamido)diquinoxal(2,3-e:2',3'-1)cyclopentanepentone. The stirred mixture was heated at 60° under nitrogen for one hour, at which time it was cooled to 0° and poured over ice, precipitating a blue-black solid. The amine salt thus formed was collected, washed with water, and air-dried. The solid material was then extracted with several portions of boiling 10% methanolic KOH, and the combined extracts filtered by suction and poured into a large volume of water. The resulting suspension was heated on the steam bath until all of the methanol had boiled away, leaving a blue-black precipitate of 2,3,8,9-tetraminodiquinoxal(2,3-e:2',3'-1)cyclopentanepentone. The material was collected, washed with water, and dried under reduced pressure yielding 1.5g (83%) of product.

Anal. Calcd. for, $C_{17}H_{12}N_8O$; C, 59.16; H, 3.74; N, 32.47
Found; C, 58.70; H, 3.50 N, 31.82

2,3,11,12-Tetraaminodiquinoxal[2,3-e:2',3'-1]pyrene was synthesized from the condensation of 1,2,6,7-tetraketopyrene with 1,2-diamino-4,5-(p-toluenesulfamidobenzene) followed by detosylation in concentrated sulfuric acid and ammonium carbonate treatment as reported previously (References 12 and 13).

Preparation of 1,2,5,6-Tetraaminoanthraquinone

To 200 ml of concentrated sulfuric acid was added with stirring, 20g (49 mmole) of 1,5-dinitro-2,6-diacetaminoanthraquinone. The resulting mixture was heated under nitrogen on the steam bath for one hour, at which time it was slowly poured onto 1 kg of crushed ice. The brown precipitate which formed was filtered by suction, and washed with water until acid-free. The crude 1,5-dinitro-2,6-diaminoanthraquinone thus obtained was added while wet to a solution containing 45g of sodium sulfide ($\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$) dissolved in 200 ml of water, and the mixture heated on the steam bath for one hour. During the reaction period, a solid phase reduction occurred yielding a violet precipitate of the free tetraaminoanthraquinone. The product was filtered, washed with several portions of water, and air-dried. The free tetramine was dissolved in 100 ml of concentrated sulfuric acid, and the filtered solution poured slowly onto 1 kg of crushed ice, liberating the sulfate salt of the tetraaminoanthraquinone. The precipitate was collected, washed free of acid, and then washed with several portions of 10% sodium hydroxide solution regenerating the tetramine. The product was washed with additional quantities of water, and was dried at 80° in vacuo, yielding 10g (72%) of pure, 1,2,5,6-tetraaminoanthraquinone as an amorphous maroon powder, m.p. $> 360^\circ$ (lit. m.p. $> 360^\circ$ (Reference 14)). The compound sublimes with decomposition at $400^\circ/0.05$ mm Hg.

Anal. Calcd. for $\text{C}_{14}\text{H}_{12}\text{N}_4\text{O}_2$: C, 62.68 H, 4.51 N, 20.88 M.W. = 269
 Found: C, 61.73, H, 4.32, N, 19.81

2. POLYMERS

Poly [3,9-diphenylfuro[2,3-g: 4,5-g'] diquinoxaline-2,10-diyl)-
 p-phenyleneoxy-p-phenylene]-(II)

A mixture of 2.283g (0.0100 mole) of 2,3,7,8-tetraaminodibenzofuran and 4.344g (0.0100 mole) of p,p'-oxydibenzil was stirred and flushed with nitrogen for 15 minutes, and then 200 ml of freshly distilled m-cresol was added. The reaction mixture was stirred at 23° under nitrogen for 20 hours to give a viscous brown solution, which was precipitated into one liter of anhydrous methanol. The resultant

yellow precipitate was collected, washed with methanol, and dried under reduced pressure. The crude product was dissolved in m-cresol and reprecipitated into methanol. After washing with methanol and drying under reduced pressure at 150° for 16 hours, the polymer weighed 5.4g (92%), and had an inherent viscosity of 0.74 dl/g in concentrated sulfuric acid (0.3g/100 ml at 30°). Good films were cast from m-cresol solutions of the polymer.

Anal. Calcd, for $(C_{40}H_{22}N_4O)_n$: C, 81.34; H, 3.76; N, 9.49; O, 5.41
Found: C, 79.86, 79.77; H, 3.96, 3.83; N, 8.47, 8.70

Poly [3,9-diphenyldipyrzino[2,3-b: 2',3'-i]phenazine-2,10-diyl)-p-phenyleneoxy-p-phenylene] (III)

To 30 ml of freshly distilled m-cresol was added, under a nitrogen atmosphere, 0.3318g (0.137 mmoles) of 2,3,7,8-tetraaminophenazine and 0.5980g (0.137 mmoles) of p,p'-oxydibenzil. The mixture was stirred overnight at room temperature to produce a homogeneous dark red solution which was heated to 120°C and maintained at that temperature for six hours. The solution was then allowed to cool to room temperature and the polymer was precipitated into 500 ml of anhydrous methanol. The resulting dark red solid was collected, washed with methanol, and was dissolved in m-cresol and reprecipitated into methanol. After washing with methanol and drying under reduced pressure at 150° for 16 hours, the polymer weighed 0.79g (96%) and had an inherent viscosity of 0.32 dl/g in concentrated sulfuric acid (0.3g/100 ml. at 30°C).

Anal. Calcd. for $(C_{40}H_{22}N_6O)_n$: C, 79.85; H, 3.51; N, 13.96
Found: C, 78.92; H, 3.95; N, 13.10

Poly [(15-oxo-3,10-diphenyl-15H-dipyrzino[2,3-g: 2',3'-g']cyclopenta[1,2-b: 3,4-b']diquinoxaline-2,11-diyl)-p-phenyleneoxy-p-phenylene] (IV)

To 20 ml of de-aerated m-cresol was added under nitrogen 0.500g (1.45 mmole) of 2,3,8,9-tetraminodiquinoxal(2,3-e,2',3'-i)cyclopentane-pentone and 0.630g(1.45 mmole) of p,p'-oxydibenzil. The mixture was stirred overnight at room temperature, during which time only slight

evidence of reaction became apparent. The reaction mixture was therefore heated at reflux for an additional eight hours, affording a dark, homogeneous solution. The polymer solution was precipitated into 500 ml of methanol, and the resulting dark solid was collected, washed with methanol, and air-dried yielding 0.820g of crude product. The polymer was further purified by reprecipitation from m-cresol into methanol. The purified material was collected, washed with methanol, and dried under reduced pressure. The polymer exhibited an inherent viscosity of 0.45 dl/g in m-cresol, and 0.06 dl/g in concentrated sulfuric acid (decomposition).

Anal. Calcd. for $C_{45}H_{22}N_8O_2$; C, 76.48; H, 3.14; N, 15.86
Found; C, 74.93; H, 3.01; N, 14.73

Poly [8,18-diphenylpyrazino[2,3-i]pyrazino[2",3": 6',7']quinoxalino [2',3': 9,10]phenanthro[4,5-abc]phenazine-7,19-diyl)-p-phenyleneoxy-p-phenylene] (V)

To 100 ml of freshly distilled m-cresol was added, under a nitrogen atmosphere, 0.2845g (0.6098 mmoles) of 2,3,11,12-tetraaminodiquinoxal [2,3-e,2'3'-l]pyrene and 0.2649g (0.6098 mmoles) of p,p'-oxydibenzil. The mixture was heated at the rate of 3°C/min. to 120°C and maintained at that temperature for 18 hours. The solution was then allowed to cool to room temperature and the polymer was precipitated into 700 ml of anhydrous methanol. The dark red solid material was dissolved in methane sulfonic acid and reprecipitated into anhydrous methanol. After washing with methanol and drying under reduced pressure at 150° for 16 hours, the polymer, 0.50g (98%), had an inherent viscosity of 0.43 dl/gm in concentrated sulfuric acid (0.3g/100 ml at 30°C).

Anal. Calcd. for $(C_{56}H_{28}ON_8)_n$; C, 81.14; H, 3.40; N, 13.51
Found: C, 80.03; H, 3.10; N, 13.02

Poly[2,10-diphenyldipyrzino[3,4-*c*: 8,9-*j*]-5,12-dioxoanthracene-3,9-diyl)-*p*-phenyleneoxy-*p*-phenylene] (VI)

To 25g of de-aerated *m*-cresol was added 0.5000g (1.86 mmole) of 1,2,5,6-tetraaminoanthraquinone and 0.8081g (1.86 mmole) of *p,p'*-bis(phenylglyoxaloyl)diphenylether. The resulting mixture was stirred overnight at room temperature under nitrogen, and then heated for eight hours at reflux, affording a dark, homogeneous solution. The polymer solution was then precipitated into 500 ml of methanol, and the resulting solid polymer was collected, washed with methanol, and dried under reduced pressure. The polymer exhibited an inherent viscosity of 0.43 dl/g in *m*-cresol.

Anal. Calcd. for $C_{42}H_{22}N_4O_3$: C, 79.99 H, 3.52 N, 8.88
 Found: C, 78.13, H, 3.40, N, 7.92

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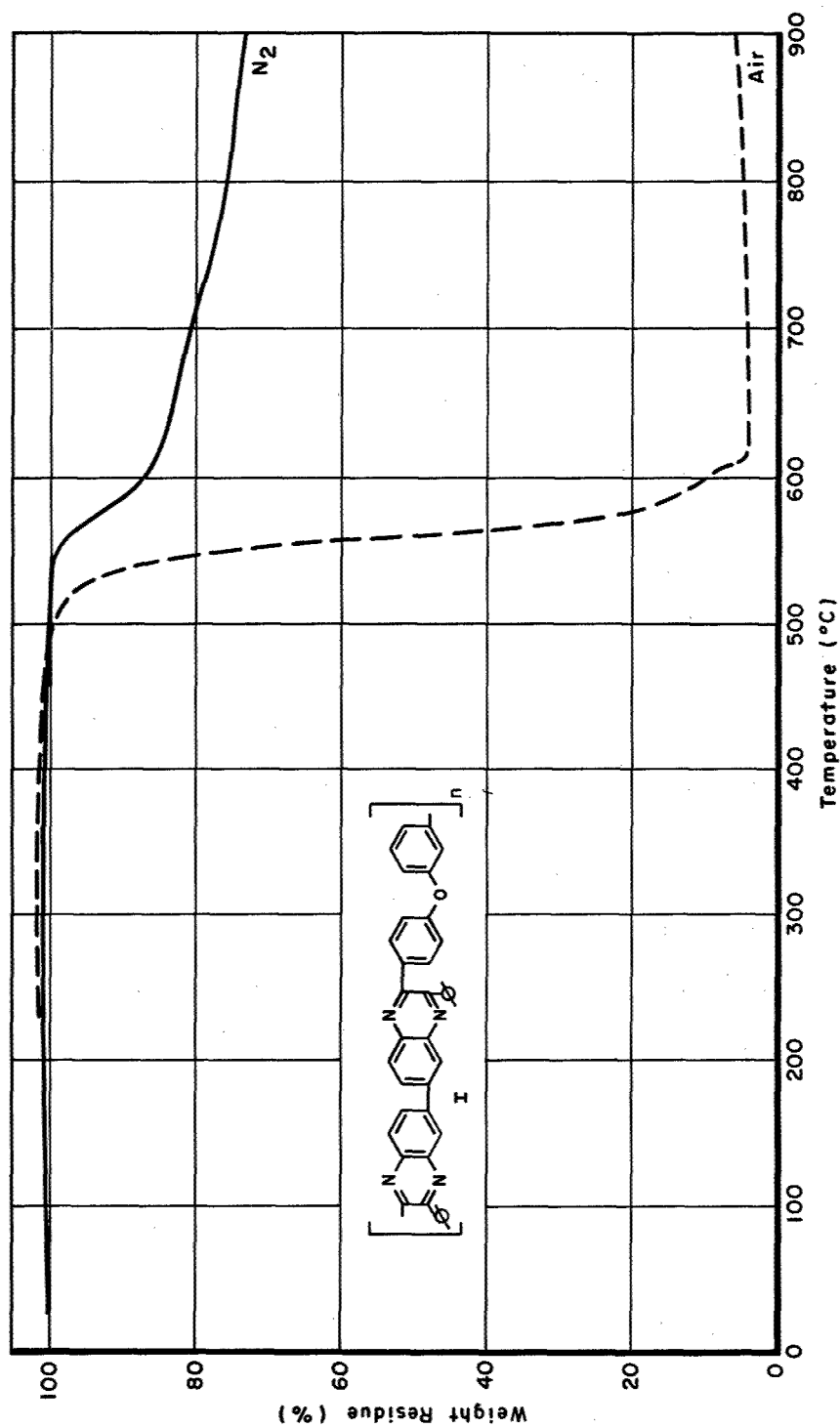
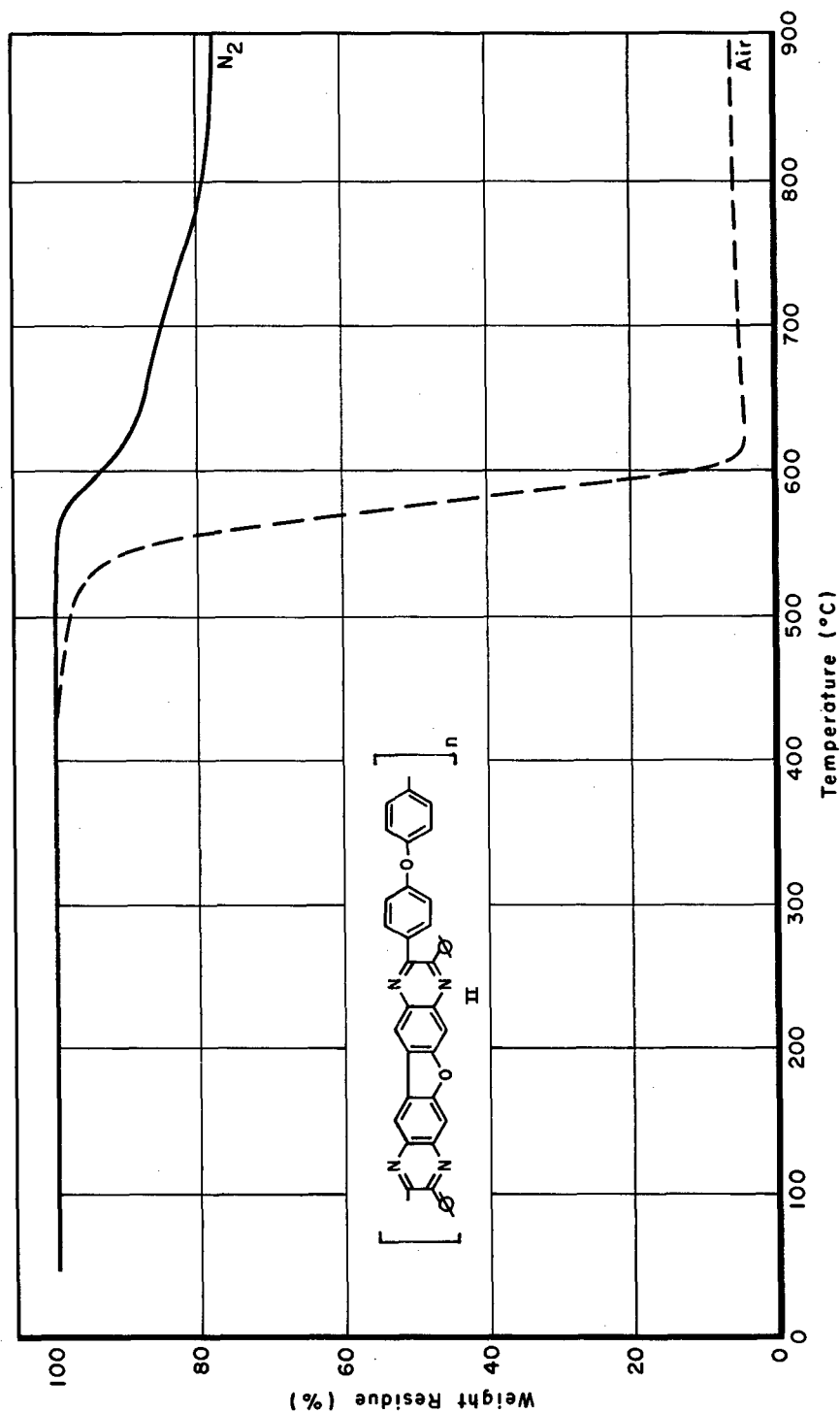


Figure 1. TGA of Poly [2,2'-(4,4'-diphenyl ether)-6,6'-bis(3-phenyl-1-quinoxaline)] (1)



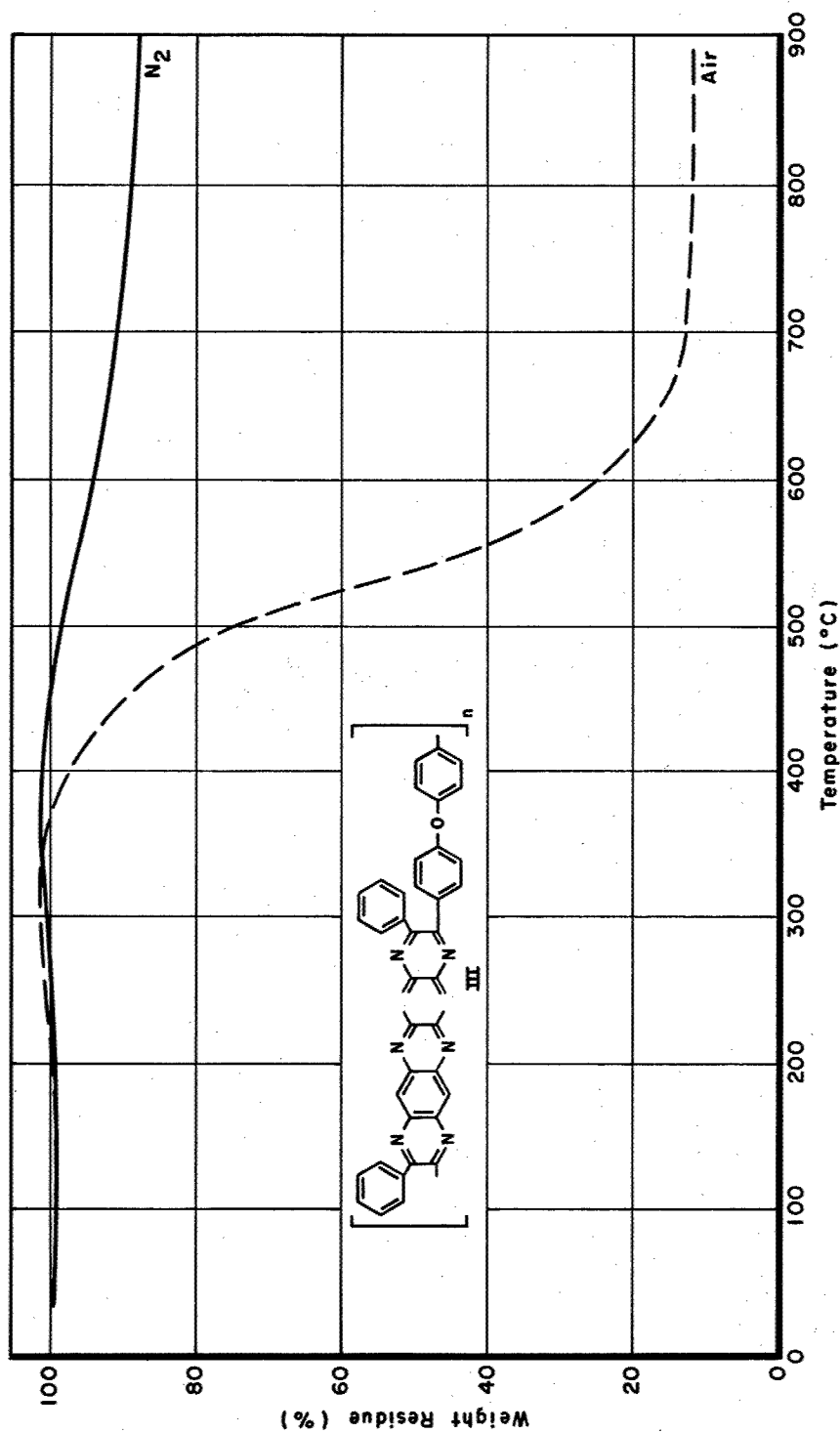
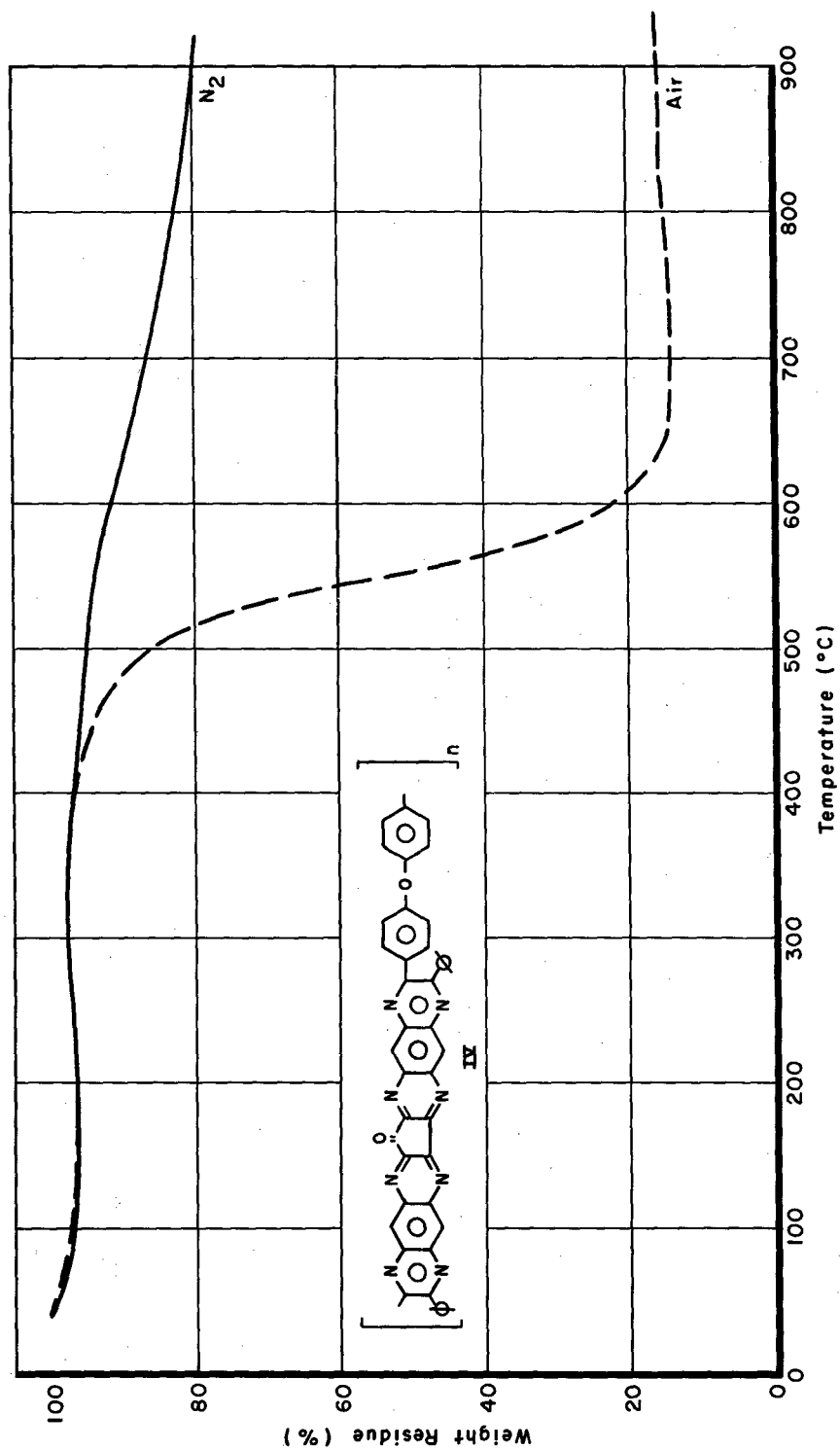


Figure 3. TGA of Poly[(3,9-diphenyldipyrazino[2,3-b:2',3'-i]phenazine-2,10-diyl)-p-phenyleneoxy-p-phenylene] (III)



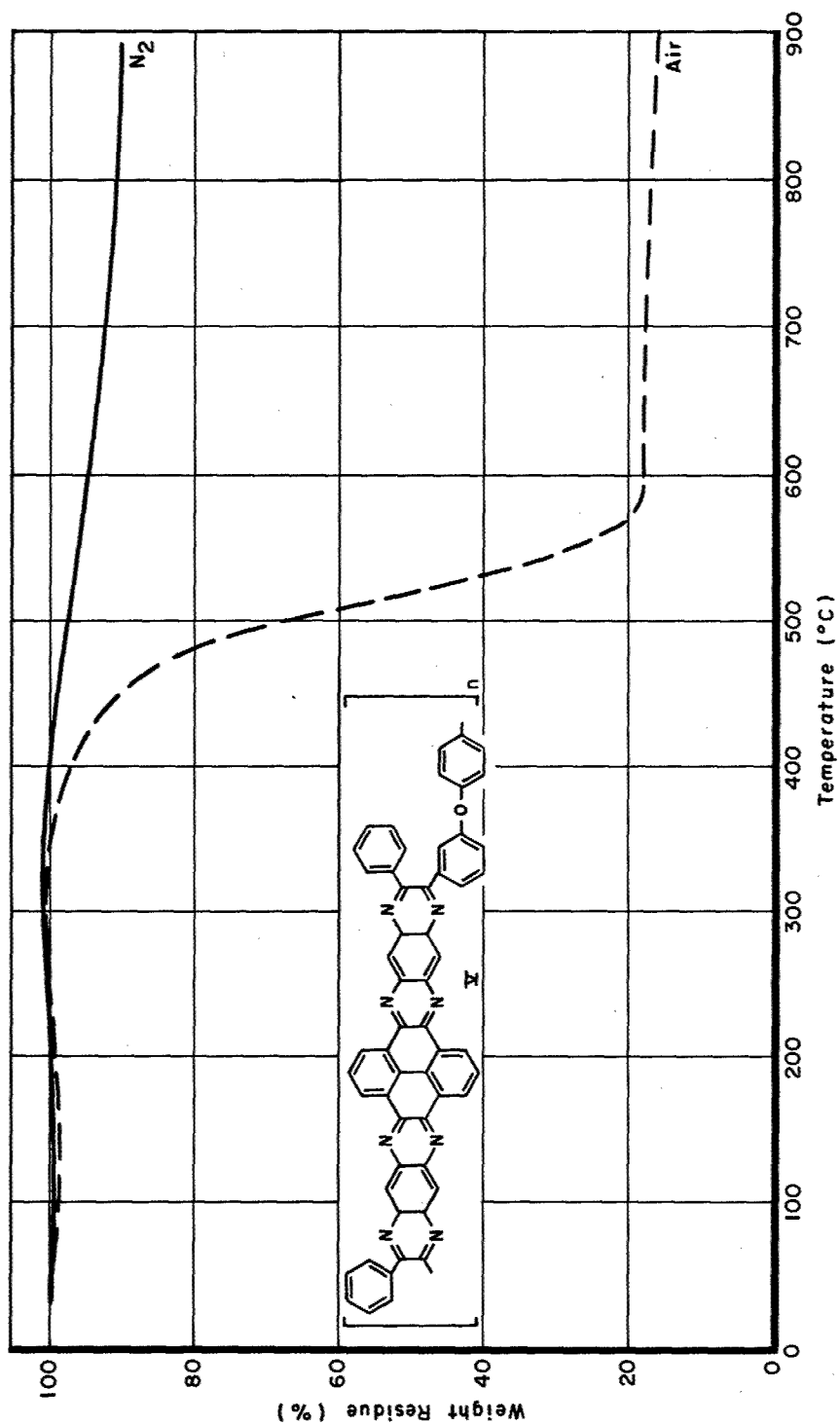


Figure 5. TGA of Poly [(8,18-diphenylpyrazino[2,3-i]pyrazino[2'',3'': 6',7']quinoxaline [2',3': 9,10]phenanthro[4,5-abc]phenazine-7,19-diy1)-p-phenylene] (V)

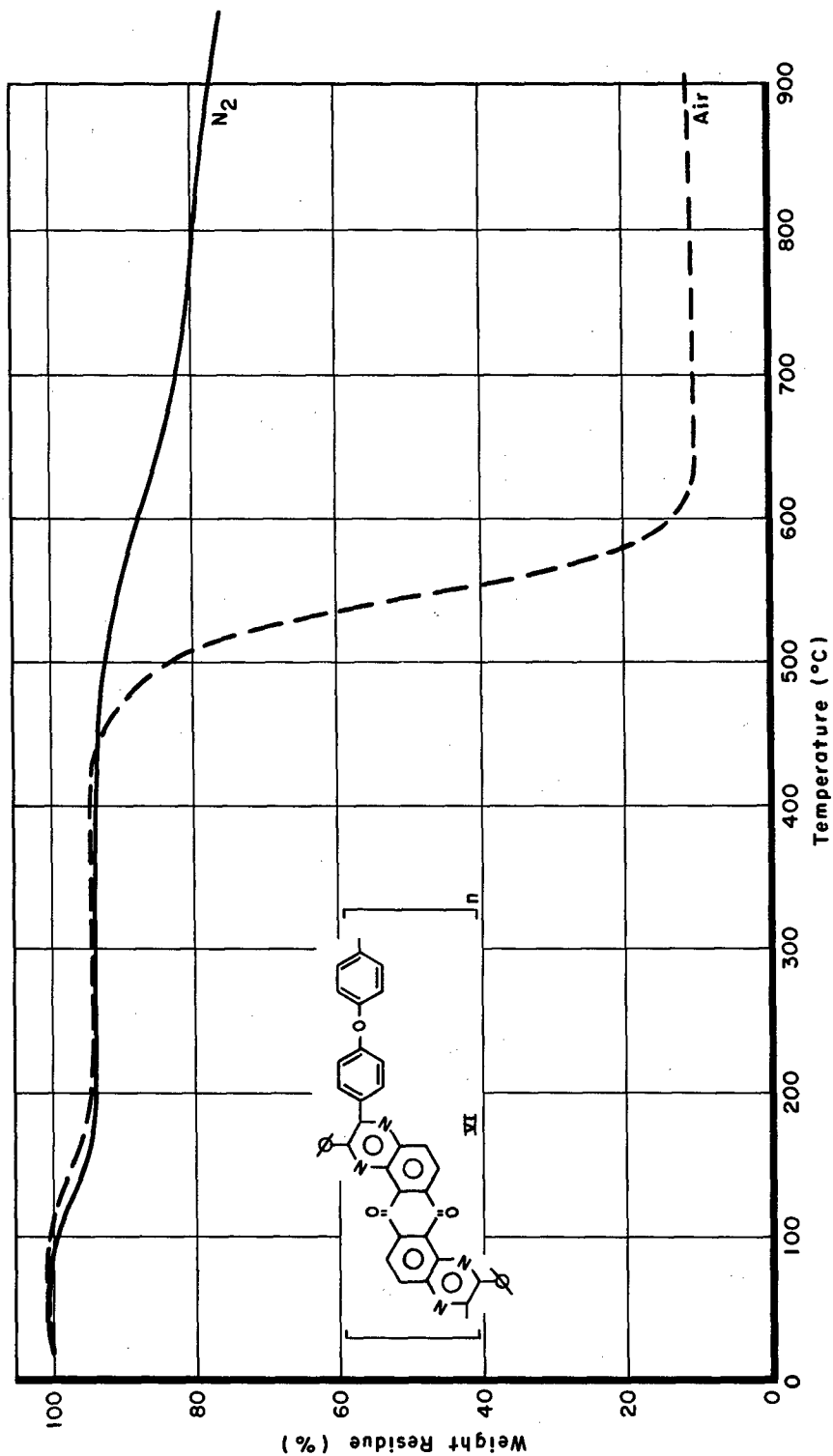


Figure 6. TGA of Poly [(2,10-diphenyldipyrzino[3,4-c: 8,9-j]-5,12-dioxoanthracene-3,9-diyl)-p-phenylene] (VI)

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| 13. ABSTRACT <p>A series of polyphenylquinoxalines has been synthesized by the reaction of p,p'-oxydibenzil with aromatic tetraamines containing varying numbers of fused rings. In addition, it was found that the glass transition temperatures of the resultant polymers increased as the number of adjacent fused rings in the polymers was increased. This provided the basis for achieving glass transition temperatures for the polymers in excess of 400°C, substantially higher than the state-of-the-art polyphenylquinoxalines. All of the polymers displayed both the good thermal stability and the solubility in <u>m</u>-cresol characteristic of the polyphenylquinoxalines family of polymers.</p> | | | |

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| 14. | KEY WORDS | LINK A | | LINK B | | LINK C | |
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| | | ROLE | WT | ROLE | WT | ROLE | WT |
| | Polyphenylquinoxalines High-Tg Polymers Thermally Stable Polymers | | | | | | |

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